Synthesis of M-type SrFe$_{12}$O$_{19}$ by mechanosynthesis assisted by spark plasma sintering

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ABSTRACT

We present a comparative study of synthesis of M-type strontium hexaferrite from strontium and iron single oxides mechanically activated by high-energy ball milling for 5 h, and assisted by two different methods: (a) conventional heat treatment and (b) Spark Plasma Sintering (SPS), both at relative low temperatures ($\leqslant$900 °C). Although it was found that both methods promoted the complete structural transformation of precursors to Sr-hexaferrite phase (S.G. *Pmc21*) for temperatures above 700 °C, higher saturation magnetization was found for SPS samples. X-ray diffraction analysis revealed that the structural transformation undergoes formation of an intermediate metastable structure (Fe$_2$Sr$_2$O$_5$) in both methods, but with slight different kinetics. Maximum specific magnetization of 67 emu/g at 18 kOe and coercivity of 3.7 kOe were recorded from powder mixtures milled for 5 h, which were subsequently SPS-ed at 700 °C. By contrast, although magnetization values for the same milled samples after being annealed at 700 °C slightly decreased, it leads to a significant increase in the coercive field reaching 5.4 kOe. These results are explained on the basis of factors such as the complete formation of hexaferrite and the attained density of consolidated powders, in addition to particle and grain sizes also reported.

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1. Introduction

M-type hexagonal ferrites have been extensively used as permanent magnets, high-density magnetic recording media [1] for the last decades and are currently used in microwave devices working at frequencies in the gigahertz range [2]. Although many other hard magnetic materials have been developed after these years, the hexaferrite performance/cost ratio is still extremely favorable. The unit cell of these of ferrites consists of a spinel block (S) with two layers of four oxygen atoms with three divalent metal ions between each layer in four octahedral sites, where the cation is surrounded by six oxygen anions and two tetrahedral sites where four oxygen anions surround the cations; and a block (R) (with the stoichiometry (SrFe$_6$O$_{11}$)$^{-2}$) with three hexagonally packed layers of four oxygen atoms each, but one of the oxygen atoms in the center layer is replaced by a similarly sized divalent metal atom with an overlap of hexagonally and cubically packed layers [3].

Among these ferrites, strontium hexagonal ferrite, with strontium as divalent metal ion, SrFe$_{12}$O$_{19}$, possesses a special place

due to its magnetic properties and particularly due to its larger magnetocrystalline anisotropy [4].

Hexaferrites can be synthesized by several processes. The conventional and oldest one is by calcination and sintering of a mixture of oxides or carbonates in a furnace at 1300 °C [5]. This process produces large particles and consumes extensive energy. Nanostructured hexaferrites can be produced by different methods such as sol–gel [6,7], hydrothermal [8], coprecipitation [9], spray-drying and microemulsion [10], conventional route [11] among others [12,13]. A particular method is mechanosynthesis; typically this method promotes formation of ferrites by mechanical activation of carbonate strontium and iron oxide [13,14]. In comparison with the traditional method (solid state reaction) [15], the mechanochemical method has demonstrated to achieve high coercivity and magnetic saturation in these materials [11–16], nevertheless the mechanosynthesis is a potential process for mass production.

Luo [14] prepared strontium hexaferrite by mechanosynthesis of a mixture of SrCO$_3$ and Fe$_2$O$_3$ with subsequent annealing. They obtained an amorphous material after ball-milling for 30 h, and a SrFe$_{12}$O$_{19}$ single phase after annealing at 900 °C for 2 h. The attained saturation magnetization reached 58.2 A m^2/kg (58 emu/g), whereas the coercivity was 281.2 kA/m (3500 Oe) at room temperature. A similar study using the mechanosynthesis

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route was reported by Sharma et al. [16] to obtain barium hexaferrite. By setting up the same experimental parameters they reported certain enhancement in some magnetic properties; such as the saturation magnetization and coercivity. Other authors like Ketov et al. [17] studied the effect of mechanochemical treatment and subsequent annealing of $SrFe_{12}O_{19}$ powders. From their results it can be inferred that even when the milling action cause deterioration on the magnetic properties, the annealing process led to a sharp improvement of them, due to crystallization and formation of the $SrFe_{12}O_{19}$ phase with fine crystallites. The best magnetic properties achieved for the milled hexaferrite were reached ($\mu_0 H_{ci} \approx 0.42$ T, $B_r \approx 0.24$ T and $(BH)_{max} \approx 9.6$ kJ/m^3) after the milling and annealing actions of the powder at 950–1000 °C for 1 h. In this case, the average particle size was about 1 μm and the average crystallite size was 100–200 nm.

Although there is a great number of published works in the magnetic materials processing via the mechanosynthesis technique, and thus showing its technological importance, this technique itself seems not to provide enough energy as to promote the complete formation of hexaferrite, even when using long milling times. It is for this reason that a post-heat treatment must be applied to the milled powder to complete formation of the strontium hexaferrite. Thus, one approach to succeed in this task is to apply annealing temperatures larger than 750 °C, although it leads to an increase in the particle size. In order to find out technical alternatives to synthesize and sinter strontium hexaferrite, with no substantial grain growing [17], we propose to apply the Spark Plasma Sintering (SPS) technique to the oxide powder mixtures ball milled for 5 h. The SPS process is known as a conveniently superfast and low temperature route not only useful to consolidate, but also to promote the chemical reaction of nanoparticles. Briefly, in the SPS method [19], the sample (usually powder) is compressed into a graphite dies matrix while operated in vacuum conditions and high intensity electric pulses are supplied into the system. Electric current goes therefore through the die, which allows heating rates as high as 1000 °C/min. If the sample is a conductor, the electric flux goes also through the sample leading to a more efficient heating process. Recent experimental results show that in addition to the heating process, the electric flux promotes ionic diffusion in the sample, thus resulting in an enhanced sintering process. But even when the sample is a non-conductive electric material, it appears that the electric field can allow the diffusion processes [20]. Comparatively, the conventional heat treatment or annealing is a long time process, which is also based on atomic diffusion, but due to usually prolonged treating times, an increase in both particle and crystallite size is expected when used as a method for synthesize [21].

In this work, we compared two different processing routes for strontium hexaferrite, both starting from commercial oxides high-energy ball milled for 5 h. The first series of materials was conventionally annealed from 700 to 900 °C while the other, consisted of SPS-ed powder at temperatures as low as 700 °C. As it will be shown, both routes induced the complete formation of nanostructured strontium hexaferrites, but with different magnetic properties, as a consequence of the different particle size.

2. Experimental procedure

Fe_2O_3 (Sigma Aldrich, 99% purity) and calcinated SrO (Sigma Aldrich, 99.9% purity) powders were used as precursor materials. These powders were mixed in a stoichiometric ratio to obtain hexaferrite according to the following equation:

$$SrO + 6Fe_2O_3 \rightarrow SrFe_{12}O_{19} \qquad (1)$$

A total of 5 g of the starting mixtures were loaded with steel balls of 1.27 cm in diameter inside a steel cylindrical vial (50 cm^3) (steel/steel, S/S) at room temperature. The mixture was milled using a high-energy ball mill Spex 8000D to promote a mechanical activation of the sample. The milling time was set to 5 h in order to avoid the sample contamination from the milling medium [13]. The ball to powder

weight ratio was 10:1. To prevent excessive heating of the vials, experiments were carried out by alternating 90 min of milling followed by 30 min in standby. All experiments were performed in argon at room temperature. After that, the milled powder (activated for 5 h) was post-treated in order to synthesized the strontium hexaferrite by following two routes: (a) compaction at 800 MPa and annealing from 500 to 900 °C for 2 h in air atmosphere, using a tube furnace and (b) SPS at different temperatures with a heating rate of 100 °C/min using a Dr. Sinter 1050 apparatus, applying pressure of 635 MPa and 6×10^{-2} Pa vacuum, setting holding times at 500, 600, 700, 800 and 900 °C for 10 min.

All the obtained compacts were characterized by X-ray diffraction (XRD) using an Equinox 3000 equipped with a multichannel detector (X'celerator) and by using Co Kα_1 ($\lambda = 1.7854003$ Å) radiation. Patterns were collected in a 2θ interval of 20–85° with increments of 0.03 (2θ). A qualitatively particle size analysis was performed by using a scanning electron microscopy (SEM) JEOL JSM 6300.

Magnetization studies were carried out at room temperature using a MicroSense EV7 vibrating sample magnetometer The coercivity (H_c), specific magnetization (M) and magnetic polarization (J) data were measured, with a maximum applied field of 18 kOe (1400 kA/m).

3. Results and discussion

3.1. Characterization of the crystalline structure

Fig. 1 shows the X-ray diffraction (XRD) pattern of stoichiometric mixtures milled for 5 h and annealed at different temperatures, from 500 to 900 °C. The XRD pattern corresponding to the mixture of powders as-milled for 5 h (Fig. 1), exclusively shows peaks of Fe_2O_3 (ICSD 22505, *R3cH*). SrO diffraction peaks are not apparent in XRD patterns because of stoichiometry, Sr concentration is 12 times smaller than that of Fe, and in this figure, the whole diffraction pattern is strongly compressed. Crystallite sizes calculated for as milled powders are around 29.4 nm, confirmed by the wider peaks on XRD pattern.

An increase in grain sizes was calculated after annealing temperature of 600 °C up to 39.5 nm and an increase in microstrain up to 0.0014. This increase in microstrain could indicate proximity of a phase change. At this temperature, it is interesting to remark the appearance of a new peak at 33.9° of 2-theta. This phase is identified as $Fe_2Sr_2O_5$ (ICSD 66403, *Ibm2*) which is a metastable structure between Fe_2O_3 and hexaferrite that acts as a nucleation site for the formation of hexaferrite. At temperatures between 600 and 700 °C new peaks corresponding to the hexaferrite are detected ($SrFe_{12}O_{19}$, ICSD 16158, *P63mmc*), but peaks corresponding to hematite still remain, indicating that the reaction has not been completed.

At 700 °C microstrain still increases for hematite structure, a value of 0.0032 is achieved, but hexaferrite structure starts to
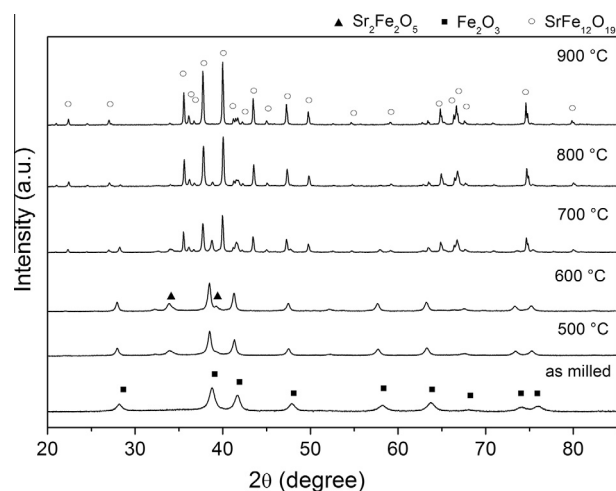

Fig. 1. X-ray powder diffraction patterns of mixture (Fe_2O_3 + SrO) milled for 5 h, pressed at 800 MPa and annealed at indicated temperatures, from 500 to 800 °C.
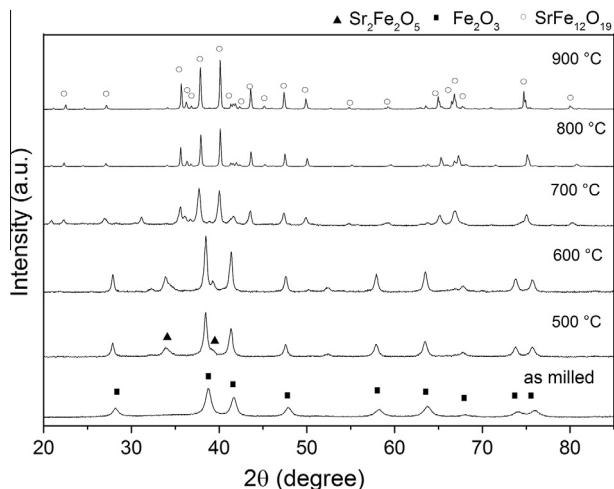
Fig. 2. X-ray powder diffraction patterns of mixture (Fe_2O_3 + SrO) milled for 5 h and submitted to SPS at different temperatures, from 500 °C to 900 °C (compacts).

Fig. 3. SEM micrograph of powder milled for 5 h.

appear, and volume percent was calculated of 78% for $SrFe_{12}O_{19}$ structure.

For temperatures higher than 800 °C, only the diffraction peaks belonging to the strontium hexaferrite, $SrFe_{12}O_{19}$, are identified, assuring the completion of the reaction (Eq. (1)). The comparison with the as-milled mixture of oxides shows an increase in grain size, indicated by the narrowing of peaks, and a diminution in microstrain for hexaferrite structure, associated to the crystallization and crystal growth of the milled powders. At this temperature the peak corresponding to the metastable phase has disappeared. Besides, a crystallite size of 95.1 nm (0.0017 of microstrain) for compacts annealed at 800 °C was calculated by Rietveld refinement from XRD pattern.

The XRD patterns corresponding to mechanically activated mixtures for 5 h and submitted to SPS at different temperatures, from 500 to 900 °C are shown in Fig. 2. In contrast to annealing treatment, faster reaction mechanism is observed by SPS. As it can be appreciated, after SPS at 500 °C a decomposition of milled Fe_2O_3 has started, increasing its microstrain from 0.00035 to 0.00157, and showing the formation, as in the previous technique, of an intermediate phase of distrontium diiron oxide III ($Fe_2Sr_2O_5$, *Ibm2*). Then, when SPS is applied at 600 °C, the milled Fe_2O_3 reduces its microstrain down to 0.00064, and the $Fe_2Sr_2O_5$ disappears. At 700 °C of SPS, new diffraction peaks corresponding mainly to the hexaferrite are detected and the peaks corresponding to Fe_2O_3 and the metastable phase mentioned diminish. After 700 °C only peaks corresponding to $SrFe_{12}O_{19}$ are detected. A crystallite size of 70 nm (0.0025 of microstrain) for compacts SPS'ed at 700 °C was calculated by Rietveld refinement from XDR pattern, the obtained value is lower than the crystallite size of the compact sintered by annealing, 95.1 nm.

3.2. Morphological characterization

As it is observed from Fig. 3, for milling time of 5 h, the powder morphology is generated by aggregates of irregular and rounded particles; qualitatively it is possible to infer an average particle size of 300 nm. It is known that the particle size is one of the most important parameters that modify the coercivity of the magnetic materials, and its effect depends of the transition from the single domain to multidomain state. The critical diameter of the particle separating both magnetic states for $SrFe_{12}O_{19}$ is 367.22 nm [23]. Therefore, the strontium hexaferrite obtained by mechanosynthesis assisted process is expected to exhibit single domain behavior.
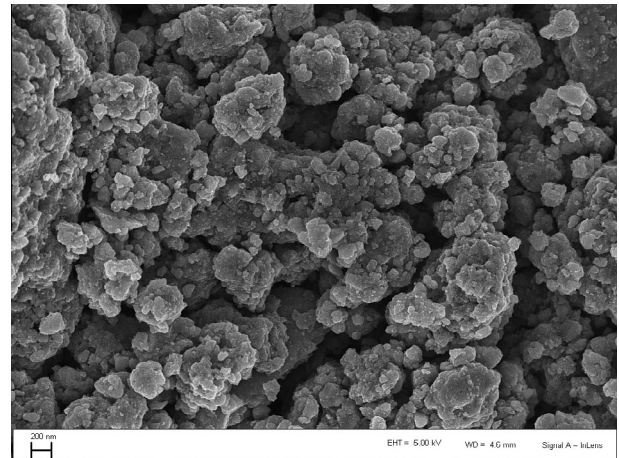
3.3. Magnetic properties

The magnetic hysteresis loops ($J–H$) of samples mechanically activated (for 5 h), pressed at 800 MPa and subjected to annealing treatment at different temperatures, are shown in Fig. 4. As it can be observed, at temperatures lower than 700 °C, the synthesis has not produced a significant quantity of hexaferrite and the magnetization is close to zero. In contrast, at temperatures higher than 800 °C, the hexaferrite is completely synthesized and the heat treatment has not detectable effect on the hysteresis loops. The magnetic properties of the obtained materials $J \sim 0.30$ T at 1440 kA/m ($M \sim 58$ emu/g) and $H_c \sim 506$ kA/m ($\sim$6.4 kOe) are in agreement with permanent magnet values of nanostructured $SrFe_{12}O_{19}$ synthesized by different methods [13–23].

The effect of the temperature of SPS in the magnetic hysteresis loop is shown in Fig. 5. In agreement with the XRD results previously presented in Fig. 3, the mixture milled for 5 h and SPS at 600 °C shows a very small value of polarization ($\sim$0.05 T) associated with the magnetic behavior of α-Fe_2O_3, and high coercivity ($\sim$300 kA/m) related to the presence of very small grains of hexaferrite ($SrFe_{12}O_{19}$), since the transformation to hexaferrite has just started [24]. Only a small amount of hexaferrite has been formed. Increasing the SPS temperature up to 700 °C, the value of specific saturation magnetization at 1440 kA/m increases up to $\sim$0.32 T (67 emu/g), a value slightly higher than the reported for this type
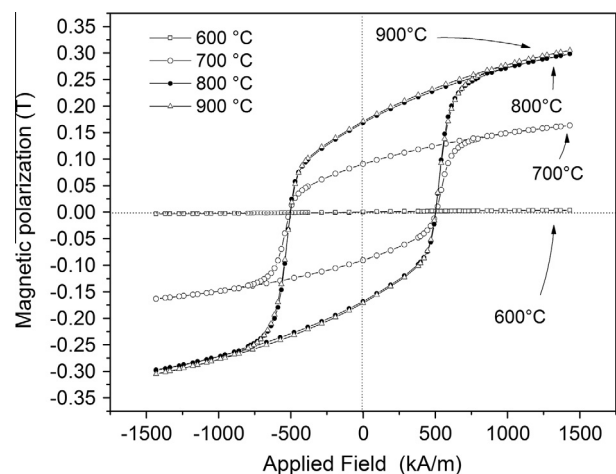

Fig. 4. ($J–H$) Hysteresis loops of precursor's mixtures pressed at 800MP, assisted by annealing at different temperatures, from 600 to 900 °C (compacts).
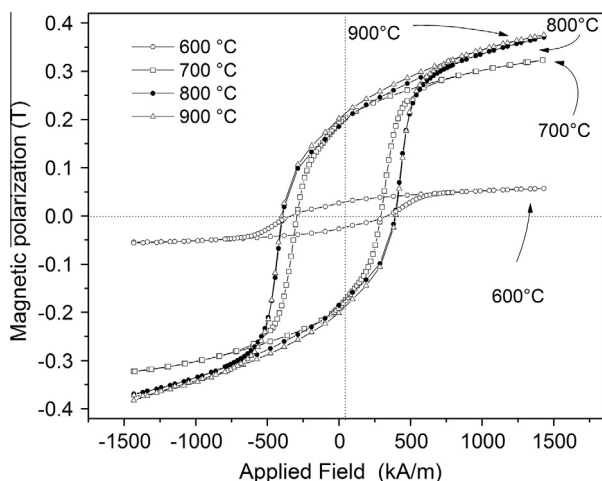
Fig. 5. (*J–H*) Hysteresis loops of precursor's mixtures assisted by SPS from 500 to 900 °C (compacts).

Fig. 7. Magnetization as a function of $1/H^2$ of precursor's mixtures milled for 5 h and assisted by (a) annealed at 800 °C (square spot) and (b) SPS at 700 °C (circle spots).

of hexaferrite [25], indicating its complete formation. The coercivity is about 286 kA/m (3.6 kOe), notably lower than the reported. By further increase of temperature up to 900 °C, a similar effect is produced, independent of temperature, an increase in the polarization (magnetization) takes place, achieving 0.37 T (65 emu/g) and also, an increase in coercivity, 391 kA/m.

A comparative analysis between two studied routes is presented in Fig. 6. It is shown the magnetic hysteresis loops (*J–H*) of the mechanically activated mixtures for 5 h and treated by (a) annealing at 800 °C, and (b) treated by SPS at 700 °C. As it can be observed the hysteresis loops reveal that in both cases saturation is not reached at the maximum applied field studied (1440 kA/m) and in comparison with the theoretical field for an anisotropic magnet, this value is very close to results recently reported [26]. The SPS method leads to a higher value of polarization (at the maximum applied field), which can be attributed to a complete transformation into the hexaferrite phase than the samples annealed at 800 °C (~0.32 versus ~0.29 T, respectively). In contrast, the coercive field exhibits an opposite behavior, i.e., it is larger in the annealed samples than in the SPS ones, which is attributed to the different particle size.

Besides, it can be observed in Fig. 6 that the coercivity H_c varied with the synthesis conditions, reaching 508 kA/m (6.4 kOe) for the
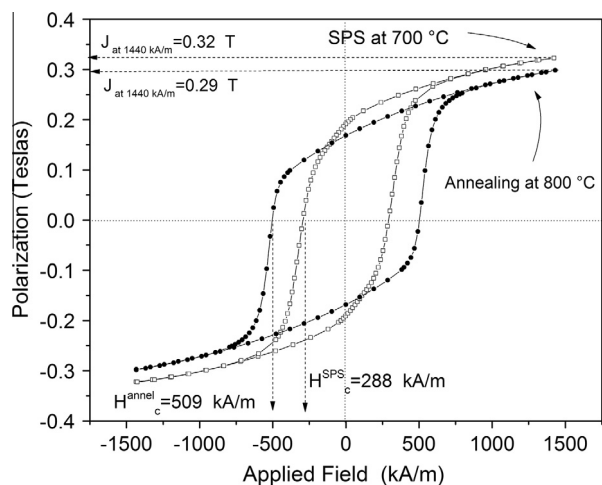
sample annealed at 800 °C, which is nearly twice that of the coarse grain counterpart treated by SPS at the same temperature. The H_c is a microstructure-sensitive parameter, which depends on grain shape, size, distribution, etc. For multidomain materials, coercivity generally depends on an inverse manner on grain size; as grain size decreases, H_c increases, and it reaches a maximum for the transition towards the single domain structure. If grain size is further reduced, H_c decreases and eventually disappears in the superparamagnetic state. These differences can therefore be attributed to the lower grain size, since SPS is a fast sintering method that reduces the atomic diffusion at high temperature. When the material exhibits a single domain behavior, the coercivity (H_c) is linearly related to the particle size [27]. Consequently, it is concluded that by decreasing particle size below the single domain limit, the coercivity is decreased [28].

These magnetic properties indicate that the M-type strontium hexaferrite magnets synthesized by SPS process can be applied in electromagnetic fields wave absorption and/or magnetic recording due to low H_c and almost constant M_s value (or J_s) [26,27].

In order to corroborate that the material is in the single domain state, an estimation of the saturation magnetization based on the Stoner–Wohlfarth (SW) model [29] is presented in Fig. 7. There, the variation of the magnetization (in emu/g) is plotted versus $1/H^2$ for the high-applied field range. The SW model is intended for non-interacting, single domain particles with uniaxial anisotropy and should lead to a linear relationship by plotting the measured magnetization at high fields as a function of $1/H^2$.

As it can be observed in Fig. 7, the obtained behavior shows a linear relationship between magnetization and $1/H^2$, for both treatments, and the M_s values are ~58.5 and 67.7 emu/g for the annealing and SPS samples, respectively. This matching could also suggest the single domain nature of the obtained material, following the Stoner–Wohlfarth model.

4. Conclusions

Strontium hexaferrite has been successfully prepared by two different techniques: annealing at 800 °C and SPS at 700 °C, showing similar reaction mechanisms: by annealing, a gradual formation of hexaferrite phase from a stressed hematite obtained from mechanical milling and a metastable phase, $Fe_2Sr_2O_5$ formed at 500 °C, was observed, the complete formation of hexaferrite was achieved at 800 °C; while by SPS, the same $Fe_2Sr_2O_5$ intermediate phase, was found at 500 °C of SPS treatment, then at 700 °C of

Fig. 6. (*J–H*) Hysteresis loops of precursor's mixtures milled for 5 h and (a) pressed at 800 MPa and annealed at 800 °C (circle spot) and (b) SPS at 700 °C (square spots).
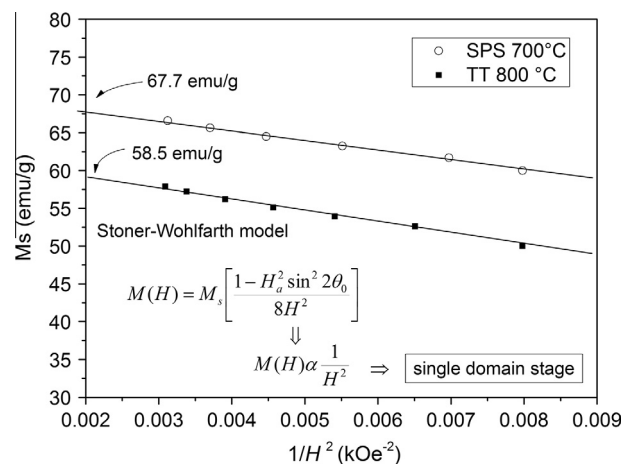
SPS, almost complete hexaferrite formation was observed, showing a faster reaction mechanism. Magnetic properties obtained for both synthesized hexaferrites (H_c = 288–509 kA/m and J = 0.29–0.32 T) were in good agreement with permanent magnet values of nanostructured $SrFe_{12}O_{19}$, synthesized by other methods. The H_c reduction and M_s increase observed in SPS samples is attributed to a lower grain size. An estimation of the saturation magnetization by the Stoner–Wohlfarth (SW) model value confirms the single domain behavior.

Acknowledgments

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